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MINERALOGICAL CONSIDERATIONS IN LEACHING OF PRIMARY COPPER
SULFIDES AT ELEVATED TEMPERATURES AND PRESSURES

David L. Leach

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MINERALOGICAL CONSIDERATIONS IN LEACHING OF PRIMARY COPPER
SULFIDES AT ELEVATED TEMPERATURES AND PRESSURES*

David L. Leach

Lawrence Livermore Laboratory, University of California
Livermore, California 94550

ABSTRACT

This report describes the leaching characteristics of four different ores in sulfuric acid systems pressurized with oxygen. The variations in the leaching characteristics between different ores can be largely attributed to differences in mineralogy. Certain gangue mineral alteration phases produced during leaching may trap copper from solution, as well as reduce the porosity of the ore. In addition, the formation of secondary copper sulfides, digenite, and covellite may limit the extraction of copper if the supply of oxygen is restricted to chalcopyrite. Apparently, the key consideration for successful leaching is maximizing the rate of oxidation of the sulfides and, at the same time, minimizing the rate of gangue mineral alteration. This can be accomplished at high oxygen pressures and moderate temperatures (70° to 90°C) and low pH (<2.0). The ideal ore mineralogy is one that is low in carbonates and easily altered Fe-Mg minerals such as biotite and hornblende, and one that has acceptable pyrite/chalcopyrite ratios.

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MASTEN

INTRODUCTION

It is inevitable that as surface deposits are depleted, copper and related metals will have to be obtained from deeper deposits. The recovery of ore minerals at depth will probably not be economically amenable to conventional mining techniques unless the ores are of significantly high grade. Consequently, it is important that we develop some new methods, such as solution mining, that will ensure a future supply of copper and similar metals.

I will restrict my discussion to the application of solution mining to relatively low-grade copper ores below the water table at a depth of 1000 ft or more. Although this type of occurrence probably contains a rather significant amount of the potential supply of copper, geologists have not been actively exploring for deep low-grade deposits largely because a technology does not exist to exploit these deposits. Considering the significant time-lag between the research and development stage and actually applying a new technology, we must proceed rapidly toward understanding the many technical uncertainties in solution mining at depth.

A proposed deep solution mining process has been under development at the Lawrence Livermore Laboratory.¹⁻⁵ The first step in such a process is the rubbleization of a region of the ore deposit by the use of nuclear explosives, or, alternatively, by undercutting and caving using conventional methods. The ore fragments are then leached in the aqueous sulfuric acid system produced by introducing oxygen into the flooded ore under hydrostatic pressure. The increase in solubility of oxygen under these conditions, together with the elevation in temperature resulting from the oxidation of the sulfide minerals, greatly accelerates the rate of copper recovery from primary copper ores.

The objective of our research program is to obtain experimental data and to develop a fundamental understanding of the leaching process that will enable extrapolation of results from laboratory experiments to field conditions. One important achievement in the LLL program was the development of a leaching model by Braun et al.³ that successfully describes the recovery of copper from ore fragments of any given particle size distribution under limited conditions of temperature and pH. The leaching mechanism involves mixed kinetics and includes a surface reaction within a moving reaction zone plus pore diffusion of dissolved oxygen through the reacted portion of the ore fragment to the reaction zone. The application of this model over a broad range in temperature and pH is difficult because of changes in the chemistry of the leach solution, as well as mineralogical and physical changes within the ore.

In this presentation, I will emphasize the importance that ore mineralogy, particularly the gangue mineralogy, has to the leaching of chalcopyrite ores in sulfuric acid solutions at elevated temperatures. The variations in the leaching characteristics that we observe between different ores can be largely attributed to differences in mineralogy and to a lesser degree, the grain size of the mineral constituents. For a given set of experimental conditions (T, PO_2 , pH), the mineralogy of the ore controls the composition of the leach solution, and the amount of alteration products yielded during leaching. These factors in turn have important controls on the recovery of copper.

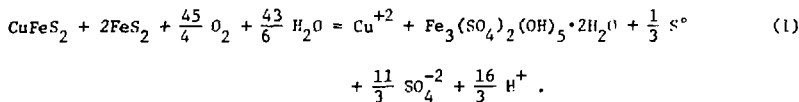
EXPERIMENTAL LEACHING OF PRIMARY SULFIDE ORES

Braun et al.³ described a series of experiments demonstrating that a porphyry copper ore can be successfully leached at 90°C and 400 psig oxygen with deionized water as the starting solution. The ore used in these

experiments is a primary porphyry copper ore from the San Manuel Mine (Magma Copper Company, Arizona). The general mineralogy of the San Manuel ore is given in Table 1.

The copper recovery for one of these experiments is shown in Fig. 1. For the experiments at 90°C, the pH of the system decreased from an initial value near neutral to a steady-state value near 2 (Fig. 2). The buffering effect observed in these experiments is an important feature of the leaching system and represents a steady-state balance of H^+ -dependent reactions involving both the sulfides and gangue minerals.

The net chemical reaction of the sulfide minerals in laboratory leaching San Manuel ore is:



The hydrogen ions produced in this reaction are mostly consumed either by reaction with calcite to produce calcium sulfate or by reaction with other gangue minerals to release equivalent amounts of other cations, principally Mg^{+2} and Al^{+3} . The system is observed to buffer at a quenched pH of about 2, representing a steady-state balance of H^+ -dependent reactions.

The chemical composition of the leach solution for a self-buffered experiment at 90°C is shown in Fig. 2. The high sulfate concentration is an important feature of the system. Most of the metal ions produced during leaching are combined with sulfate as metal-sulfate complexes or precipitates.

The calcium concentration shown in Fig. 3 steadily decreases because of the precipitation of anhydrite. The Na, K, Fe, and Al concentrations are

limited by the precipitation of jarosite-alunite minerals $[\text{KF}_2(\text{SO}_4)_2(\text{OH})_6 - \text{KAl}_3(\text{SO}_4)_2(\text{OH})_6]$ with Na substitution for K and limited substitution between Al and Fe].

Jarosite and alunite are the most common alteration products observed in the leached samples. They occur as fine-grained material throughout the rock and as replacements of K-feldspar and K-mica.

Leaching experiments at temperatures in the 70° to 150°C range and at pH values in the 0.25 to 2.0 range clearly show that temperature and pH have a pronounced effect on the rate and ultimate recovery of copper from San Manuel ore. These effects are predictable in part from the leaching model developed by Braun et al.³ Their model is based upon a diffusional rate constant for oxygen and a chemical rate constant, both of which are temperature dependent. Although the leaching model does not directly account for variations in pH, several factors that are included in the model are affected by pH. For example, the porosity of the ore would be greater at a low pH because of less precipitation of iron salts.

At temperatures of 90°C or less, the effect of maintaining a pH below the self-buffered value of approximately 2.0 is to increase the rate of extraction, particularly in the early stages of leaching. The effect at 70°C is shown in Fig. 4. The increase in rate of extraction may be caused by the increased oxidation of chalcopyrite by Fe^{+3} or by the increase in porosity of the rock because of less precipitation of alteration products.

At 110°C, successful recovery can be accomplished only if the pH is between approximately 2.0 and 0.75. At temperatures in excess of 110°C, leaching is unsuccessful at any pH.

The leached ore from the experiments was studied by x-ray diffraction, optical microscopy, and electron microprobe analyses. The results show that

physical and mineralogical changes in the gangue and sulfide minerals are major limiting factors, particularly where certain mineral reactions are accelerated at elevated temperatures. Decomposition or alteration of the silicate minerals to copper-bearing phases are accelerated at elevated temperatures, particularly when high temperatures are combined with low pH. Elevated temperatures also accelerate the rate at which chalcopyrite is replaced by secondary sulfides, covellite, and digenite.

Leaching experiments were conducted on three other ores, including two ore types from the Ruth Mine (Kennecott Copper Corporation, Ruth, Nevada). One is a typical porphyritic, highly altered quartz monzonite referred to as Ruth. The other is a hornfels-type, fine-grained rock consisting largely of plagioclase and hornblende. This ore will be referred to as Ely. The third ore is from the Kelley Mine (Anaconda Company, Butte, Montana). The general mineralogy of the ores is given in Table 1.

The extraction rates for San Manuel, Ruth, and Kelley at 90°C, 400 psig O₂ (Fig. 5) are very similar, which is not surprising considering the similar mineralogy and texture of the ores. However, at 110°C the extraction curve of Kelley ore was quite different from that of San Manuel under the same conditions. At 110°C, the Kelley curve passed through a maxima of 80% extraction at 160 days, then decreased to approximately 45% extraction at the termination of the experiment at 280 days. The copper that had been successfully leached from the ore was incorporated into silicate alteration products yielded during leaching.

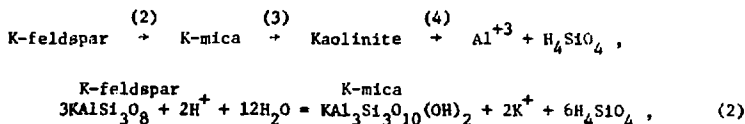
The Ely ore could not be successfully leached at any temperature or pH. The extraction curve for Ely at 90°C and 400 psig O₂ is shown in Fig. 6. The curve passed through a maxima of 20 to 30% extraction, then rapidly fell to less than 5% extraction at the termination of the experiment. The Ely ore was

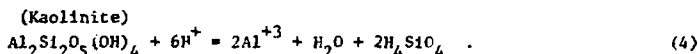
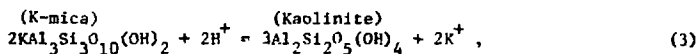
in an advanced state of alteration to a fine-ground material consisting of disaggregated rock fragments and alteration products composed of montmorillonite, amorphous phase (largely SiO_2), hematite, and jarosite. The chalcopyrite was almost completely leached, and presumably the copper entered the alteration phases. The leaching experiments on Fly and Kelley ore clearly demonstrate that the alteration of gangue minerals is an important consideration in the leaching of ores at elevated temperatures.

GEOCHEMISTRY OF LEACHING SILICATE GANGUE MINERALS

The leaching of porphyry copper ores is very similar to the hydrothermal reactions that deposited the primary sulfides. Therefore, considerable insight into the process of leaching sulfide ores can be obtained from the extensive body of research on hydrothermal ore deposition.^{6,7} For the leaching experiments, the T, PO_2 , and pP are fixed. The pH is controlled by the addition of sulfuric acid or by the production of H_2SO_4 from the oxidation of the sulfides and is buffered largely by the $\text{HSO}_4^-/\text{SO}_4^{--}$ boundary as well as related equilibria.

The process by which the gangue mineral reacts with the acid solution may be defined as "hydrolysis" or "base leaching." This process can be viewed as a decomposition reaction with water in which H^+ is consumed and an equivalent amount of cations released to the solution. This type of reaction between the silicate minerals and the aqueous solution containing H^+ ions is perhaps best illustrated by the reaction:





Reactions to the right consumes H^+ ions and releases an equivalent amount of K^+ for Eqs. (2) and (3) and of Al^{+3} for Eq. (4). Since the activity of the solids and water may, to a first approximation, be taken as unity and the H_4SiO_4 concentration fixed by the equilibrium with amorphous silica, the equilibrium constant at a given temperature and pressure takes the simple form:

$$K = \frac{a\text{K}^+}{a\text{H}^+} \quad \text{Eqs. (2) and (3)}$$

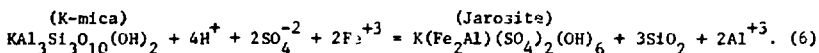
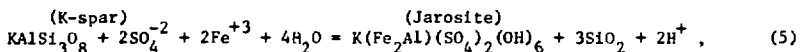
and

$$K = \frac{a\text{Al}^{+3}}{(a\text{H}^+)^3} \quad \text{Eq. (4) .}$$

The lack of thermodynamic data on the chemical species in the leach solution at the temperatures of interest prevents calculation of the activities from concentrations. However, one can make an "order of magnitude" approximation of the relative stability of the gangue minerals by equating concentrations with activities. Using the analyses of the leaching solutions and the data of Helgeson,⁸ it is clear that K-spar, K-mica, and kaolinite are grossly out of equilibrium with the leach solution. Similar argument can be made to show that, with the exception of quartz, all of the major gangue minerals should be decomposing. Petrographic studies on the leached ores show that the relative reactivity of the gangue minerals in acid solution is:

Hornblende > Biotite > Plagioclase > K-spar > K-mica .

The K^+ and Al^{+3} concentrations are much lower than what is expected because of precipitation of a jarosite-alunite phase. Rather than releasing K^+ to the solution as in a simple hydrolysis reaction, the K^+ along with H^+ , $SO_4^{=}$, and Fe^{+3} may be fixed at K-rich sites (K-spar, K-mica, and biotite). The most apparent alteration of K-spar and K-mica can be shown as:



Hornblende, biotite, and plagioclase alteration products generally consist of a very fine-grain brown clay together with a jarosite-alunite phase. The brown clay is believed to be a mixture of montmorillonite, jarosite-alunite, anhydrite, hematite, and a glass phase (probably amorphous clay and SiO_2).

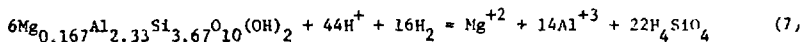
At temperatures of 90°C or less, the rates of these reactions are rather small compared with the rate at which the sulfides are oxidized. At temperatures greater than 110°C, the rate of silicate gangue mineral decomposition is greatly accelerated. This is important because some of the alteration phases may trap copper as distinct copper phases or else by adsorption.

POSSIBLE COPPER BEARING ALTERATIVE PHASES

The abrupt loss of copper from solution observed in several leaching experiments, particularly with Ely ore, may be caused by the incorporation of copper into certain alteration products. Petrographically, the only unique feature of the leached ore in these experiments is the rather advanced state

of gangue mineral alteration. There is an appreciable amount of jarosite-alunite, montmorillonite, an amorphous phase ($\approx 60\% \text{SiO}_2$), and hematite.

Montmorillonite: The formation of montmorillonite is of considerable interest, both because it is capable of trapping copper and because it is a common alteration product in the leached ore. The copper can be adsorbed on the clay mineral surfaces, occur as an interlayer cation, or occur in a lattice position substituting for Al (medmontite⁹). The activity product constant for a hypothetical Mg-Montmorillonite from Eq. (7):



is

$$K_{(T)} = \frac{(\text{Mg}^{+2}) (\text{Al}^{+3})^{14} (\text{H}_4\text{SiO}_4)^{22}}{(\text{H}^+)^{44}}$$

Helgeson⁸ gives us the following values for $\log K_{(T)}$:

Temperature, °C	25	60	100	150
$\log K_{(T)}$	36.60	16.49	-0.96	-18.25

At a constant pH, the significant decrease in $\log K$ with an increase in temperature shows that the formation of montmorillonite is favored at high temperatures.

Because montmorillonite is very fine grained and dispersed with other alteration products, we have not been successful in identifying a distinct copper montmorillonite. It is reasonable, however, to suspect that copper may be adsorbed onto the clay particles. Significant amounts of copper do commonly occur in the fine-grained alteration products including montmorillonite.

Amorphous Phase: An amorphous phase is a common alteration phase and is particularly abundant in the leached ore that showed significant loss of copper during the course of the experiment. X-ray analysis indicates approximately 60% of the amorphous phase is SiO_2 . The remaining is probably amorphous clay material. Electron microprobe analyses did not detect any copper in the glass phase. However, if copper was adsorbed on the surface of the amorphous phase, preparation of the sample for analysis perhaps removed it from the surface.

Jarosite-Alunite: X-ray diffraction studies have shown the presence of either alunite ($\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$), jarosite ($\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$), and possibly natroalunite ($\text{NaAl}_3(\text{SO}_4)_2(\text{OH})_6$). Minor amounts of copper were detected by electron microprobe analysis of a jarosite-alunite phase in ore leached at high temperatures (110°C). However, no copper was detected in the jarosite-alunite phase formed at 90°C or less. It is possible that high temperatures may lead to the formation of a phase similar to one of the alunite minerals, beaverite ($\text{Pb}(\text{Cu},\text{Fe},\text{Al})_3(\text{SO}_4)_2(\text{OH})_6$). The similar ionic size of Cu^{+2} (0.69\AA) and Fe^{+3} (0.64\AA) may permit substitution if valence compensation is made elsewhere in the structure.

Altered Biotite: Electron microprobe analysis has shown the presence of minor amounts of copper to be located at sites of altered biotite crystals. It is not clear whether the copper is located within the leached biotite (vermiculite phase) or in the associated montmorillonite.

Formation of Secondary Sulfides: Covellite and digenite were observed in all the leached ore; they can be found in experiments that have leached well and in those that have leached unsatisfactorily. During leaching, covellite and digenite are continually being formed in the interior of the ore fragments as copper diffuses into the rock and reacts with the chalcopyrite. As the oxidizing zone advances into the ore fragment, chalcopyrite, as well as

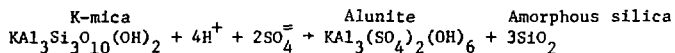
the previously formed secondary covellite and digenite are oxidized and release copper to the solution. As long as the rate of oxidation in the reaction zone proceeds faster than the loss of copper to form digenite and covellite, the ore will have a favorable leaching rate.

This is particularly important for the success of an in-situ leaching operation because if for some reason the supply of oxidant is restricted to part of the rubblized ore, this part of the ore will act as a "sink" for copper successfully leached elsewhere.

OTHER CONSIDERATIONS

Acid Consumption: For ores with relatively high carbonate content, it may be necessary to add sulfuric acid to the leach solution to maintain a sufficiently low pH (near 2.0). This is particularly true in the early stages of leaching where the dissolution of calcite is most rapid. For San Manuel ore, acid additions were unnecessary because the amount of sulfuric acid produced from the oxidation of the sulfides was sufficient to dissolve the calcite. However, Kelley, Ruth, and Ely ores required additions of acid to maintain the desired pH during early stages of leaching. It is desirable to have a reasonable amount of pyrite relative to the chalcopyrite content because of the increase in production of sulfuric acid. Conversely, too much pyrite leads to higher oxygen consumption. Therefore, the calcite content of the ore, as well as the pyrite/chalcopyrite ratio are important considerations in evaluating the economics of solution mining. In addition, the dissolution of calcite $\text{CaCO}_3 + 2\text{H}^+ + \text{SO}_4^{=}$ $= \text{CaSO}_4 + \text{CO}_2 + \text{H}_2\text{O}$ liberates carbon dioxide causing a reduction in oxygen partial pressures either by dilution or by stripping oxygen from the leach solution.

Volume Change: Because chemical and mineralogical changes occur within the ore during leaching, there will be changes in mass and in volume (pore volume as well as fracture volume). During the course of leaching, basic cations and CO_2 are removed from the ore whereas H^+ , O_2 (in the form of $\text{SO}_4^{=}$), and H_2O are added to the ore. The overall change in mass during leaching may be small when compared with the change in volume of the ore. The formation of the common alteration phases, alunite, jarosite, amorphous phase, montmorillonite, and hematite have significantly larger molar volumes than do the primary silicates from which they formed. The alunitization of K-mica as shown below can lead to a significant increase in volume.



\bar{V} = molar volume

$$\bar{V} \text{ alunite (146.8 cm}^3\text{)} + 3\bar{V} \text{ SiO}_2(27.27 \text{ cm}^3\text{)} - \bar{V} \text{ K-mica (140.71 cm}^3\text{)} \\ \text{(amorphous)}$$

$$\Delta \bar{V} \text{ reaction} = +87.9 \text{ cm}^3$$

(maximum percent increase in volume = 62%)

The gangue minerals seldom are completely altered; however, they do react sufficiently to reduce the pore and fracture volume of the rock, particularly at high temperatures. The effect of reducing the porosity of the rock is to restrict the supply of oxygen to the sulfides. This in turn lowers the copper extraction rate.

SUMMARY

The experimental and theoretical work conducted at LLL demonstrated that chalcopyrite ore can be successfully leached in sulfuric acid systems pressurized with oxygen. We believe that this technology can be used successfully for the in-situ recovery of copper and that this method is particularly attractive for ore deposits that cannot be economically mined by conventional methods.

We showed that the gangue mineralogy plays an important role in the rate and ultimate recovery of copper. The temperature and pH of the leach solution control the rate at which gangue mineral decompose to certain alteration products. The formation of alteration products may in some instances trap copper from solution, as well as reduce the porosity of the ore. Apparently, the key consideration to successful leaching is maximizing the rate of oxidation of the sulfides and, at the same time, minimizing the rate of gangue mineral alteration. This can be accomplished at high oxygen pressures and moderate temperatures (70° to 90°C), and low pH (< 2.0).

For successful leaching at elevated temperatures, the ideal ore mineralogy is one that is low in carbonates and easily altered Fe-Mg minerals such as biotite and hornblende and has acceptable pyrite/chalcopyrite ratios.

ACKNOWLEDGMENTS

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Table 1. General mineralogy of the San Manuel, Kelley, Ruth, and Fly copper ores.

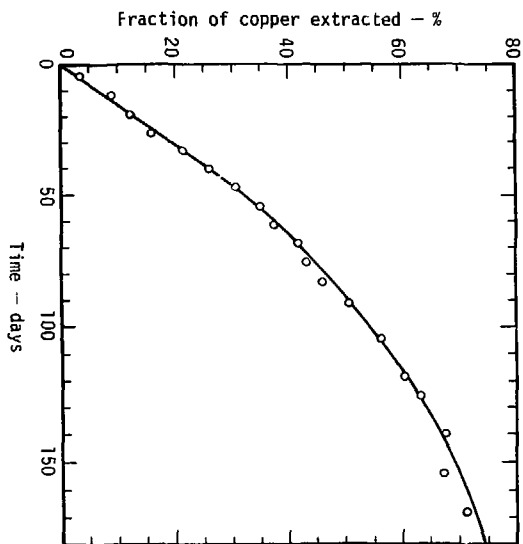
	Rock Type	Major Gangue	Minor Gangue	Sulfides
		Minerals	Minerals	
SAN MANUEL	Quartz- sericite rock	Quartz	K-spar (largely	Chalcopyrite
		K-mica	altered to	Pyrite
		Biotite	sericite)	
	Altered- quartz monzonite		Plagioclase	<u>Ore Grade:</u> 0.7 wt.% Cu
			Fematite	<u>Calcite/py</u> = 0.43
			Calcite	
			Magnetite	
	Quartz- sericite- biotite rock		Kaolinite	
			Montmorillonite	
			Hornblende	
KELLEY	Altered- quartz monzonite	Quartz	Plagioclase	Chalcopyrite
		K-spar	Chlorite	py/cp = 2.1
		Biotite	Kaolinite	Pyrite
		K-mica	Montmorillonite	<u>Ore Grade:</u> 0.49 Cu
			Calcite	<u>Calcite/py</u> = 1.38
			Hornblende	
			Zircon	
			Rutile	
RUTH	Altered- porphyritic quartz monzonite	Quartz	Calcite	Chalcopyrite
		K-feldspar	Biotite	py/cp = 0.46
		Plagioclase	Apatite	Pyrite
		Muscovite		<u>Ore Grade:</u> 1.44 wt.% Cu
				<u>Calcite/py</u> = 2.5
ELY	Altered- hornfels	Plagioclase	Calcite	Chalcopyrite
		Hornblende	Chlorite	py/cp = 2.7
		Quartz	Epidote	Pyrite
		Biotite	Apatite	<u>Ore Grade:</u> 0.75 wt.% Cu
				<u>Calcite/py</u> = 0.84

FIGURE CAPTIONS

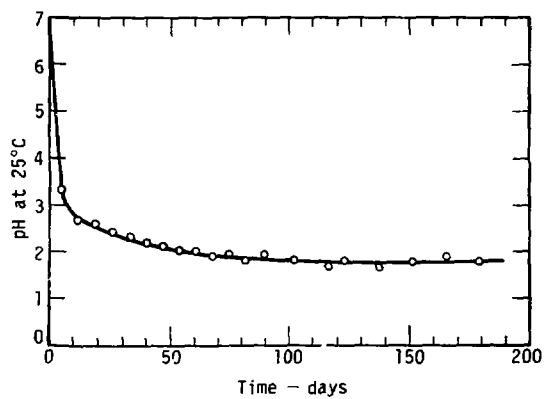
- Fig. 1. Extraction of copper at 90°C and self-buffered pH. Ore size, 16 ± 3 mm.
- Fig. 2. Solution pH during leaching at 90°C. Ore size, 16 ± 3 mm.
- Fig. 3. Chemical composition of the leach solution at 90°C and self-buffered pH.
- Fig. 4. Extraction of copper at 70°C and various pH. Ore size, 16 ± 3 mm.
- Fig. 5. Extraction rates for San Manuel, Ruth, and Kelley at 90°C, 400 psig O_2 . Ore size, 16 ± 3 mm.
- Fig. 6. Extraction curves for Ely and San Manuel at 90°C and 400 psig O_2 . Ore size, 16 ± 3 mm.

NOTICE

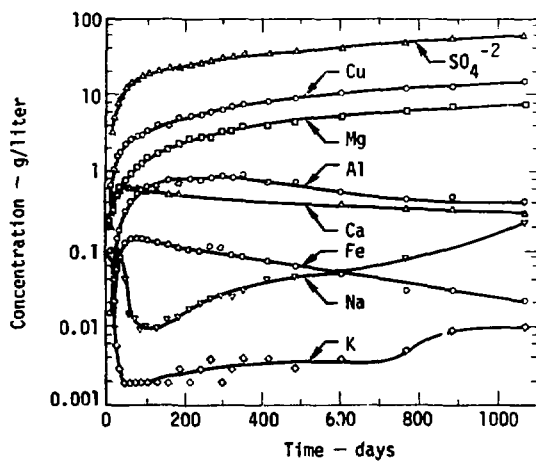
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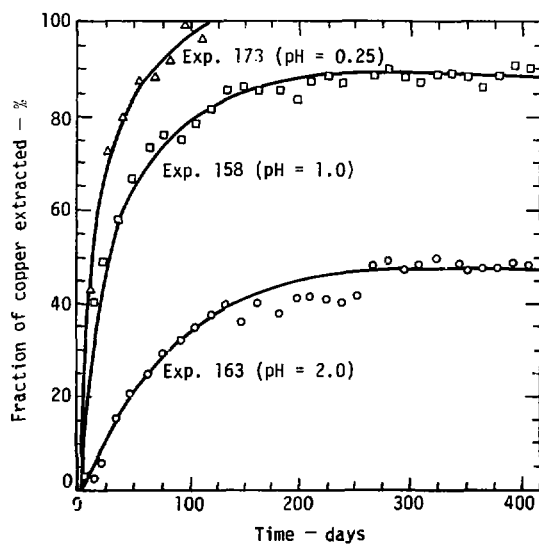
Leach - Fig. 1



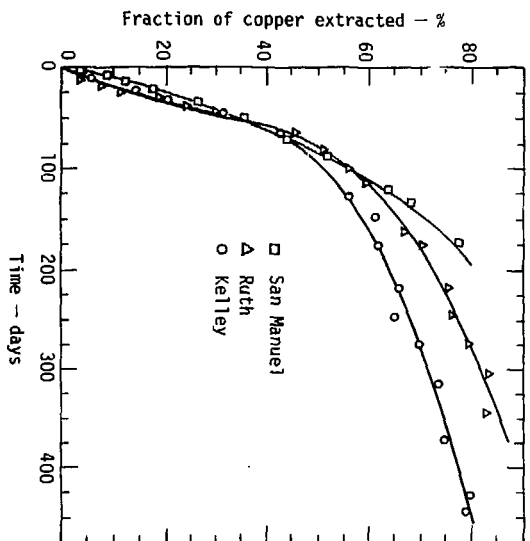
Leach - Fig. 2



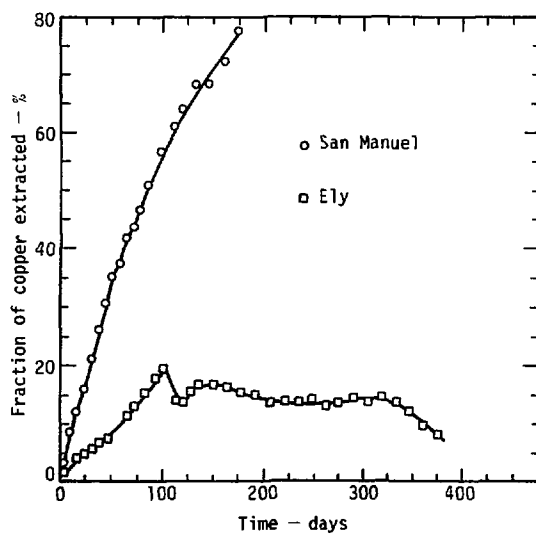
Leach - Fig. 3



Leach - Fig. 4



Leach - Fig. 5



Leach - Fig. 6